

INORGANIC CHEMICAL PRECIPITATE FORMATION
PAYLOAD DESIGN

by Craig Friedrich, Ph.D.
Department of Mechanical Engineering
Louisiana Tech University

ABSTRACT

The ever increasing use of the microgravity environment in materials research prompted the investigation of the formation of inorganic precipitates. Sponsored by the Frontiers of Science Foundation of Oklahoma and built at Oklahoma State University, G-405 utilizes six transparent chemical reaction chambers to actively mix a dry powder with a liquid solution. At predetermined intervals the progress of the precipitate formation is photographed and stored as data. The precipitate particles will also be subject to post-flight analysis. The various tasks performed during the 14 hour duration of the experiment are initiated and monitored by a custom-built digital controller. The payload, originally scheduled and shipped for flight in 1986, is currently scheduled as the backup payload for STS-29 with a possible launch date of January, 1989.

BACKGROUND

The Frontiers of Science Foundation of Oklahoma sponsored a competition among local high school students to conceptualize a payload for space research. Mark Rubowitz and Clay Collins, two juniors at Casady School in Oklahoma City, proposed the investigation of inorganic chemical precipitate formation in the microgravity environment. Their interest in this research area was cultivated by several years of laboratory investigations and encouragement by science fair participation. The Foundation then funded the payload development at Oklahoma State University and, after a two year developmental period, the payload was ready for flight STS-61C.

CHEMICAL EXPERIMENTATION

The precipitate reactions which will be carried out by the payload are shown below. The first compound is the dry powder for that chamber, the second compound is the liquid for that chamber, and the fourth compound is the precipitate for that chamber.

- 1) $\text{MnSO}_4 + 2\text{KOH} \rightarrow \text{K}_2\text{SO}_4 + \text{Mn(OH)}_2$ a yellow gel precipitate
- 2) $\text{NiCl}_2 + 2\text{NaOH} \rightarrow 2\text{NaCl} + \text{Ni(OH)}_2$ a green gel precipitate
- 3) $\text{MnSO}_4 + \text{Ba(OH)}_2 \rightarrow \text{MnO}_4 + \text{BaSO}_4$ a white crystal precipitate
- 4) $\text{K}_2\text{Cr}_2\text{O}_7 + 2\text{AgNO}_3 \rightarrow 2\text{KNO}_3 + \text{Ag}_2\text{Cr}_2\text{O}_7$ a brown crystal precipitate
- 5) $\text{Hg(NO}_3)_2 + 2\text{NaCl} \rightarrow 2\text{NaNO}_3 + \text{HgCl}_2$ a white curdy precipitate
- 6) $\text{NaCl} + \text{AgNO}_3 \rightarrow \text{NaNO}_3 + \text{AgCl}$ a white curdy precipitate

Precipitate formation first depends on the presence of a solution which is saturated with a relatively insoluble salt. If the solution is supersaturated, the salt will leave the solution as a precipitate until the solution is once again saturated. In the gravity environment this process is very dependent upon localized concentrations within the supersaturated solution. With the heavier precipitate particle settling out, the solution may be physically mixed which will aid in precipitate formation or the solution may become stratified which will create zones of precipitation similar to the nucleation sites within a solidifying metal alloy. In the microgravity environment though, the precipitate formation will be dependent only upon the localized solution concentration. This of course assumes a constant temperature throughout the solution. The data from the precipitate formation will be taken by means of photographs of the formation as well as post-flight analysis of the precipitate particles.

THE MIXING CHAMBERS

The purpose of the six mixing chambers is to allow photographic data of the particle's formation, to contain the chemical reactants, and to provide a forced mixing of the two reactants in each chemical chamber. The first two purposes are easy to accomplish through the use of relatively thick-walled plexiglas tubing and rubber O-rings. The task of force mixing the reactants became the challenge. Several active mixing schemes were addressed but all of these ideas were too complex and space consuming. Having worked as a hydraulic system designer in industry, the problem of differential volumes within an enclosed cylinder finally had a good use. In a conventional hydraulic cylinder, one side of the piston is connected to a rod which exits the cylinder body. The other side of the piston does not typically have a rod attached to it. When the cylinder

piston is moving, more fluid is being displaced on the non-rod side of the piston than on the side of the piston which contains the rod.

In the reactant chamber used for this payload it is necessary to use an arrangement which has a rod running the entire length of the cylinder. It is impractical to use a rod of varying diameter to establish the differential volume relationship, therefore this is accomplished by using two sizes of plexiglas tubing. The mixing chamber design is shown in Figure 1. The cylinder piston is shown in its preactivation configuration. Upon activation, the cylinder spring will force the piston to move and the O-ring seal between the chemical reactants will be broken. In addition the downstream chamber half has a slightly larger inside diameter than the upstream chamber half. This open annulus around the piston will allow fluid to be forced to the back side of the piston which will create the forced mixing. In tests this concept has worked with great success. An additional plus for this design is that there is only one moving part and the driving force is a spring which is compressed prior to flight, adding to the system reliability. The cylinder piston contains one O-ring seal to keep the reactants apart prior to activation. The cylinder body also has O-rings in each end to help eliminate reactant leakage into the canister interior. The cylinder body also contains absorbant material just outside of the cylinder O-rings to add an additional level of protection against reactant leakage.

To better maintain a consistent temperature within the six reaction chambers, each chamber is wrapped with two flexible resistance heaters. The heaters are each about two feet long, covered with woven fiberglass, and composed of teflon coated wires. Originally designed for use at 120VAC and 100 watts, the heaters are operated at 12VDC and put out one watt each. The heaters operate at about 90 degrees F and offer 12 watts of heat capacity at a relatively low temperature. The heaters are thermostatically controlled to become active below 60 degrees F.

SPECIAL DESIGN CONSIDERATIONS

It was necessary to design for a catastrophic failure of all six reactant mixing chambers. Several of the liquid reactants are caustics which readily dissolve aluminum. Because the entire structure is made of 6061-T6 aluminum, it was necessary to coat the structure with an inert resin. Because of the flight acceptance, PT-201 from Products Techniques, Inc. was chosen. This resin was brushed on and then baked at 360 degrees F to expedite outgassing. The coating provides a hard surface which is resistant to caustics and also provides electrical isolation of the structure. Because the flight container is also made of aluminum, it was necessary to limit the caustic concentration so that there is no possibility of the caustics penetrating the canister thickness. If all of the caustics are allowed to attack the canister interior, the wall thickness of the canister would decrease by one-thousandth of an

inch. An additional consideration in the use of the caustics is the release of hydrogen gas upon reacting with aluminum. This consideration was the overriding factor for the maximum concentration of the caustic solutions. The caustic concentration is such that if all of the caustics react with the unprotected aluminum, the maximum buildup of hydrogen gas will be less than 3% by volume.

CONTROL CIRCUITRY

The control circuitry for the operational scenario of the payload is relatively simple. Upon astronaut activation, the circuitry maintains the reactant chambers in a dormant state for one hour. During this time, the thermal environment of the chambers is stabilized by way of thermostatically controlled chamber heaters. After one hour has passed, the reactant chambers are solenoid activated and the precipitation process begins. After several ground tests, it was learned that it is necessary to record the precipitate formation early in the experiment duration. A photograph of the formation will be taken once every minute during the first 12 minutes, then once every 10 minutes for the next two hours, then once every 50 minutes for a total of 36 exposures over a 14 hour period. The control circuitry uses a simple digital clock, counters and EPROM memories to control the duration between photographs.

In conversations with Goddard Space Flight Center personnel in totally unrelated matters, it was learned that some EPROM's are so susceptible to memory damage from radiation that they have actually been used as radiation detectors. This fact led to the use of three EPROM's to provide a "majority vote" logic scheme. The 2716 EPROM was selected because of its widespread availability and its ease of programming. This 16K memory chip was programmed with a 40 byte program. Though severely under-utilized, the chip was used because of the availability of the programming hardware. The output of the EPROM's are used as the input to the voting logic. The voting logic is used to address multiplexers so that the appropriate clock pulses activate the camera shutter. This arrangement is shown in Figure 2 and the arrangement for the majority voting logic circuitry is shown in Figure 3.

PREVIOUS FLIGHT SCHEDULING

G-405 was originally scheduled as first backup payload for STS-61C for a March 1986 flight. To meet this schedule, it was necessary to ship the completed payload to KSC in November of 1985. The payload has been held in storage at KSC until early May of 1988 when it was returned to Louisiana Tech University. This provided an excellent opportunity to examine any long-term storage effects on the payload systems. All aspects of the payload withstood the 2-1/2 year dormancy very well. The payload was originally shipped void of the chemicals so the chambers and seals were still in new condition. The battery pack was the only component which showed any

signs of age. The cells used were the Gates "X" cells, each with two volts and five amp-hours capacity. The cells were combined to provide a nominal 12 volts and 20 amp-hours capacity. When shipped the batteries were charged to approximately 13 volts. After 2-1/2 years in storage, the batteries had discharged to about 11 volts but the current capacity was greatly diminished. The battery pack was capable of driving the camera autowinder only several times before being completely inadequate. After voltage tests were performed on individual cells, it was found that four of the cells were in deep discharge (less than one volt output) and were incapable of being recharged. These four cells have been replaced. All other lower voltage systems such as the flash and the digital controller were still very functional. This clearly exhibits the excellent shelf-life of the lead-acid battery.

RECOMMENDATIONS

As part of The National Space Transportation Systems's return to flight status all previously approved payloads are required to have conducted on them a Delta Phase III Review. This review requires further documentation as to tests performed on various payload systems and components. It is recommended that any tests performed as part of a hazard report verification be documented with the date and a report number. This additional work at the time of the test will save additional time and paperwork when the payload is going through the safety review process.

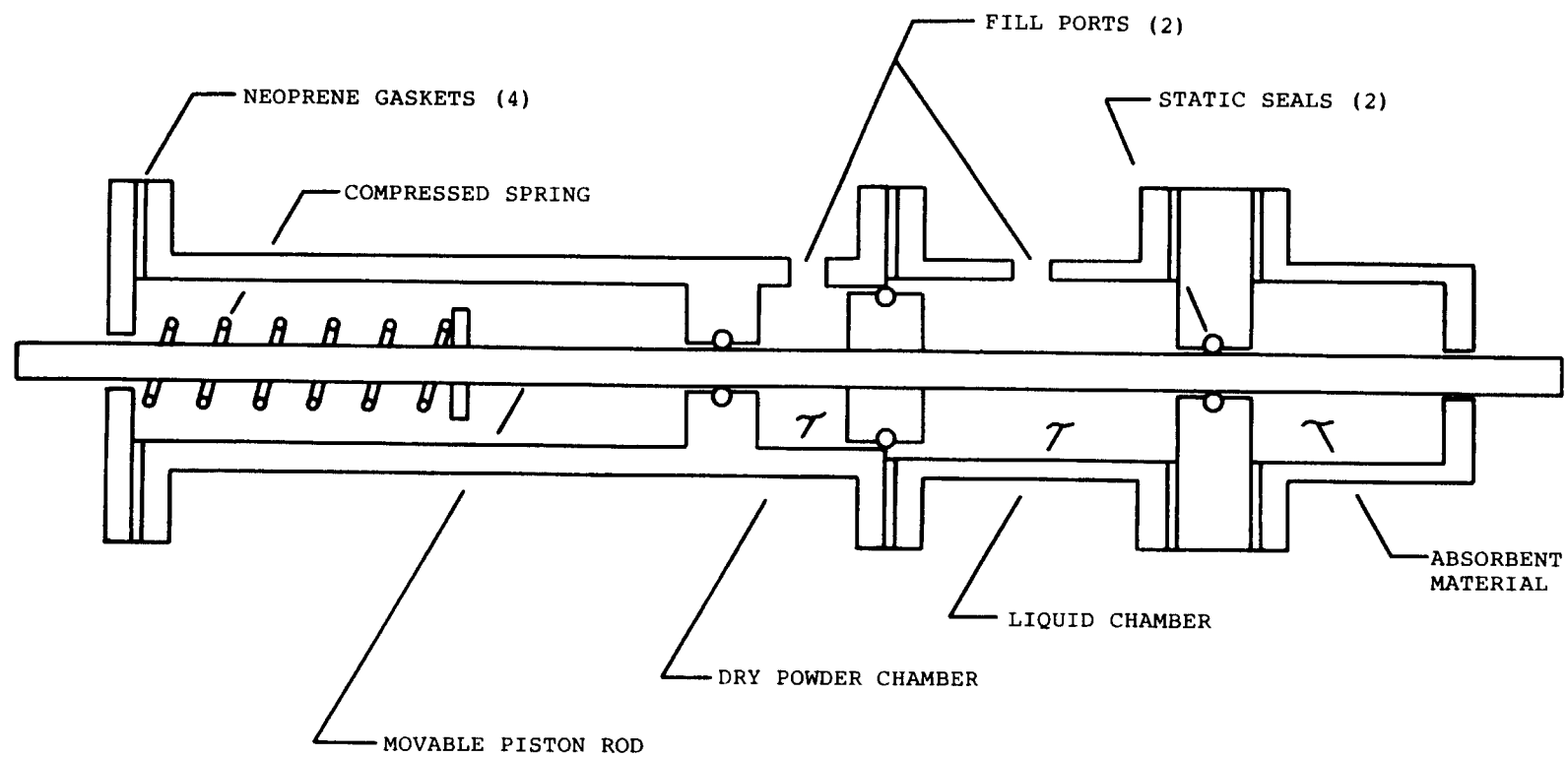


Figure 1. Precipitate Reaction Chamber

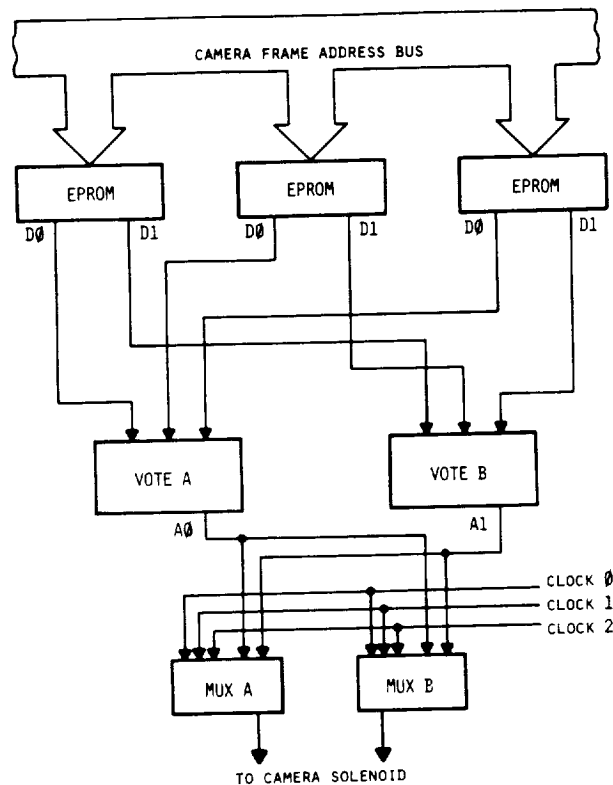


Figure 2. Camera Shutter Activation Circuitry

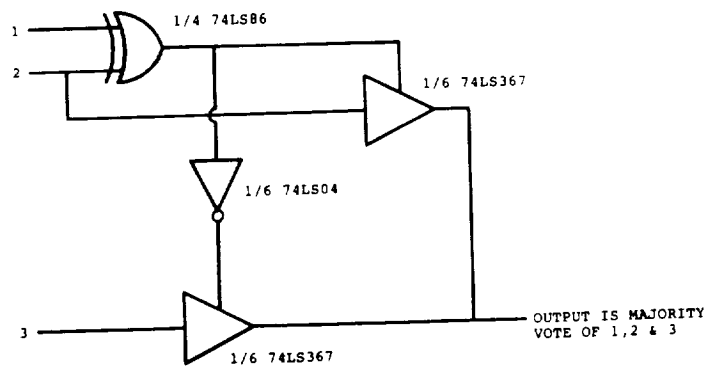


Figure 3. Majority Voting Logic Circuitry